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The Continuing Battle Against Defects in Nickel-Base Superalloys

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AGAINST DEFECTS IN NICKEL-BASE SUPERALLOYS
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THE CONTINUING BATTLE AGAINST DEFECTS IN NICKEL-BASE SUPERALLOYS

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ABSTRACT

In the six decades since the identification of age hardenable nickel-base superalloys their compositions and microstructures have changed markedly. Current alloys are tailored for specific applications. Thus their microstructures are defined for that application. This paper will briefly review the evolution of superalloy microstructures and comment on the appearance and implications of microstructural defects in high performance superalloys. It is seen that new alloys and processes have generated new types of defects. Thus as the industry continues to develop new alloys and processes it must remain vigilant toward the identification and control of new types of defects.

INTRODUCTION

The age-hardenable nickel-base superalloys may be considered to be in their sixth decade (Ref. 1). During their evolution to the modern alloys used in aircraft propulsion and other advanced turbines many changes have occurred in the alloy's chemistry, microstructure and processing. As the alloy systems have become more sophisticated, the defects in the alloys have also become more sophisticated. This paper will briefly review the evolution of microstructures of modern nickel-base superalloys and discuss the appearance and implications of selected microstructural defects in high performance alloy products.

MICROSTRUCTURAL EVOLUTION

Historical perspectives of the evolution of superalloys are available elsewhere (Refs. 1 and 2). However, it is instructive to briefly consider the

evolution of Ni-base superalloys while considering the defects in their microstructures. The evolution of the microstructures of the Ni-base superalloys to about 1970 has been described by Decker and Sims (Ref. 3) and recently extended to 1984 by Sims (Ref. 4). It is shown schematically in Fig. 1 (after Ref. 4). The earliest alloys were used as wrought products having relatively fine equiaxed grains. They contained small amounts of η (Ni_3Ti) or γ' (Ni_3Al) precipitates and carbides. One assumes that the grain size and γ'/η precipitation were controlled by forging practise and subsequent heat treatment. If the M_{23}C_6 carbides precipitated in a cellular form they were determined to be detrimental to stress rupture life and were considered a defect (Ref. 4). It was necessary to control the processing to produce a discrete M_{23}C_6 morphology at the grain boundaries.

As the superalloys evolved beyond Nimonic¹ 80A, a Ni-base alloy having Cr, Al, Ti, and C additions, the amount of γ' was initially increased by increasing the amounts of Al and Ti and later by Cb and Ta additions. Strength was further increased by refractory metal additions of Mo and W. In the mid-50's the alloys had been strengthened to a level where they were becoming extremely difficult to forge and further improvements (particularly for turbine blade alloys in the United States) required that the alloys be used in the investment cast form. Advances in vacuum melting permitted the use of greater amounts of reactive metals in the alloy compositions. In addition, vacuum melting allowed the production of cleaner materials (i.e., less oxide inclusions). As the amount of alloy additions in the melt increased and the size of billets increased, new and undesirable phases were identified. Some of these will be discussed in greater detail later.

¹Nimonic is a trade name of the INCO family of companies.

By the mid-60's the cast Ni-alloys had apparently reached their peak in stress-rupture strength, but had poor intermediate temperature ductility. This problem was addressed by alloying with Hf by the group at Martin Metals and by directional solidification (DS) by the group at Pratt & Whitney Aircraft. The latter lead to another new and distinctive class of microstructures - single crystal superalloys which were first flown in commercial jet aircraft in the early 80's. These directionally solidified products have offered a new avenue for increasing use temperature, but have their own set of microstructural defects which will be discussed later.

Also in the mid-60's the wrought alloys appeared to have reached the maximum strength levels compatible with economic forging to final product shape. Allen (Ref. 5) and his co-workers demonstrated that superalloy billets made from prealloyed powdered metal could be more easily worked to final shape and have mechanical properties comparable with the best ingot metal products. The trend toward powder metal superalloy products started and as was soon learned, new types of microstructural defects would need to be controlled. Some of these defects will be discussed in greater detail.

The evolution of Ni-base superalloys from the early Nimonic 80A, a wrought alloy of essentially four elements, to today's family of wrought from ingot, powder metal, cast and directionally cast alloys has seen many changes in the microstructures of the alloys. As the microstructures increased in complexity, and in some sense, simplicity, as in the case of single crystals, so the defects in the alloy's microstructures have also changed in character. Let us now look more closely at some microstructural defects.

SELECTED MICROSTRUCTURAL DEFECTS

General Comments

In the time allowed, it is possible to discuss only a few selected types of defects. The following sections will show examples of microstructural

defects with emphasis on newer technology materials. The examples will include additional phases, porosity, grain boundary, and grain orientation, grains in single crystals, unrecrystallized grains in an ODS alloy, and oriented precipitates in single crystal alloys. It is intended that the similarity among the same type of defects in different products might become apparent.

Additional Phases

The additional phase which, perhaps, initiated some of the greatest interest in the superalloy community is σ phase. Its identification by Wlodek (Ref. 6) as a phase deleterious to a Ni-base superalloy initiated a large volume of research and was in part responsible for the 1st International Symposium on Superalloys at Seven Springs. Figure 2 is a representative photomicrograph of σ in a Ni-base superalloy. The fact that σ may take very long times to form in some compositions and little in others as shown in Fig. 3 (Ref. 7) offered little comfort to those who wished to avoid it. The presence of the phase in large amounts could cause drastic reduction in the stress rupture life of an alloy as shown in Fig. 4 (Ref. 7).

The application of PHACOMP (Ref. 8) and similar techniques have allowed prediction of σ formation in individual heats of alloys whose broad chemical specification may be σ -prone to be identified. However, no predictive system exists which is 100 percent successful in anticipating the formation of σ , μ and similar phases in new and substantially different alloys. For example an alloy designated MMT143 which was initially RSP processed by Pratt & Whitney Aircraft has subsequently been shown to be prone to the precipitation of δ phase in cast single crystal form (Ref. 9). Its detrimental effect on stress rupture life is similar to σ in IN-100 as shown in Fig. 5.

Freckles are defects caused by segregation of elements in amounts which can cause precipitation of phases. The defect was known to forging houses in the 60's particularly in ingots which were directionally solidified. Such defects could carry through to the final product as shown in Fig. 6. A freckle in an ingot might appear as shown in Fig. 7. When directionally solidified turbine airfoils moved to production, investment casting houses learned of the freckle as shown in Fig. 8 (Ref. 10). While the effects of freckles on mechanical properties is not well documented in the open literature, one would assume that the presence of brittle phases on the surface of a product would be detrimental, particularly in fatigue behavior.

The formation of the freckle is a bit easier to follow in model systems. Work done by Hallawell (Refs. 11 and 12) using ammonium sulfate - water solutions and Pb-Sn alloys has shown that freckles result from gravity driven fluid motion which results in macrosegregation in the ingot. An example of a freckle in a Pb-Sn alloy is shown in Fig. 9. Also shown in Fig. 9 is a micrograph of a similar ingot solidified while being tilted to the gravity vector. It was rotated and precessed about the gravity vector and no freckles were observed.

Ceramic, foreign metal, and other materials may contaminate and be consolidated in powder metal products. The effect of these defects on properties was shown to be detrimental (Ref. 13), Fig. 10. The effect of these defects on the design of turbine components is reflected in Fig. 11 (Ref. 14). The detrimental effects of ceramic inclusions is also well known to the foundryman. In a study performed on a directionally solidified alloy dross and mold breakage inclusions were frequently found on the fractured surface of test bars (Ref. 10).

The Ni-base superalloys are strengthened by the γ' phase, or in a few alloys, γ'' or η phases. The lack of proper distribution of these phases

can be considered to be microstructural defects. The more advanced, highly alloyed compositions contain greater than 50 vol % γ' . It is virtually impossible to quench an engineering structure fast enough to suppress the γ' precipitation after a solution treatment. The mechanical properties of the structures are quite sensitive to the relative amounts of coarse and fine γ' present. The effect of (sub-solvus) solutioning temperature on the amount of γ' in Rene 95 is shown in Fig. 12 (Ref. 15). As the solution temperature is raised, more γ' is dissolved and becomes available for effective strengthening when it precipitates in fine form. This is particularly important to recognize when one is solution treating near the γ' solvus because the rate of change of the amount of undissolved γ' with temperature can be about 0.5 percent/ $^{\circ}$ C.

The same problem occurs in cast alloys. It is particularly apparent in single crystal alloys in which it is difficult to completely dissolve the γ' without risking melting. Fig. 13 shows an example of a PWA 1480 bar which was solution treated in a furnace intended to be slightly above the γ' solvus, but with an inadvertent temperature gradient. It can be seen that substantially different amounts of primary γ' were left in the structure. The bars with little residual primary γ' were significantly stronger in tensile tests.

The size of the fine γ' is a function of the temperature at which it precipitates and thus is sensitive to the cooling rate from the solution temperature. If the quench rate from the solution temperature is retarded, the γ' may become too large causing a degradation of stress rupture life as shown in Fig. 14 (Ref. 16). For the same materials tensile strengths were also reduced as much as 12 percent.

Porosity

Voids in cast metal products are a normal result of solidification shrinkage and the large reduction in the solubility of gasses in solids compared to liquids. Such voids, when contained within investment cast products may be called porosity or microporosity.

An example of porosity in a casting is shown in Fig. 15. It can be seen that cracks are associated with pores in this stress rupture test. Reference 10 concluded that directionally solidified castings have little tolerance for microshrinkage.

In single crystal superalloys, pores are often the site of fracture initiation in fatigue tests, Fig. 16. An apparent solution to this porosity might be the application of hot isostatic pressing (HIP) to close the pores. This has been shown to extend fatigue life (Ref. 17). However in the process of HIPing other problems may be introduced as shown in Fig. 17. In this specimen, carbon from the heating element in the autoclave is believed to have contaminated the autoclave's argon atmosphere and diffused into the carbon free single crystal alloy. The added carbon reduced the melting point allowing incipient melting to occur and the subsurface precipitation of additional phases (carbides).

Porosity is also recognized as a potential defect in powdered metal products, in particular those which receive little or no post-HIP mechanical working which tends to close some porosity. During the HIPing or other consolidation cycle, residual gasses may be entrapped in the metal. During subsequent heat treatments, these gasses may expand and cause pore formation. This is commonly called "thermally induced porosity" and specifications for "as-HIP" products typically specify a maximum permissible reduction in density for a specified thermal cycle. Pores may also be introduced by container failure during the HIP cycle. An example of such porosity is shown in

Fig. 18. Such porosity has detrimental effects on mechanical properties, Fig. 19 (Ref. 18).

Grains and Grain Boundaries

The introduction of directional solidification presented the superalloy metallurgist and designer with a substantially different structure. The new material is anisotropic, particularly with regard to elastic modulus. For application as turbine blades, the natural growth direction of the Ni-base superalloys, $\langle 100 \rangle$, also the lowest modulus direction, is essentially coincident with the blade stacking axis. This offers great benefit in terms of improved low cycle fatigue behavior for turbine blades. However, it is not immediately apparent if the improvements are due to the modulus or the lack of grain boundaries intersecting the leading or trailing edge of the airfoil. The effect of grain boundary emergent angle on thermal fatigue is shown in Fig. 20 (Ref. 19). It can be seen that when the emergent angle is low the life is significantly greater. In this experiment, the emergent grain boundary angle and the crystallographic orientation effects are confounded as shown in Fig. 21. Examination of the cracks, which failed to initiate or to propagate along the grain boundaries and were normal to the leading edge of the airfoil sample suggested that the improvement in low cycle fatigue is due to the modulus effect, Fig. 23. Thus control of the growth direction ($\langle 100 \rangle$ axis) relative to the stacking axis of the turbine blade is imperative. Reference 10 found that the emergent, convergent and divergent grain boundary angles had only minimum effects on mechanical properties of DS alloys.

Oxide dispersion strengthened (ODS) alloys resemble DS products in the sense that they contain elongated grains. It is commonly thought that a high grain aspect ratio is requisite for the development of optimum mechanical properties. Figure 23 shows an example of Incoloy² alloy MA6000 which was

²Incoloy is a trade name of the Inco family of companies.

improperly processed. A small volume of material was found to be chemically different from the bulk and failed to properly recrystallize. Attempts to determine the effect of this defect on mechanical behavior were inconclusive. It is suggested that such a defect, particularly near the surface could be detrimental to fatigue life.

The single crystal superalloy is metallurgically a less complex system than that of the DS superalloy. To take full advantage of the single crystal form, the elements B, C, and Zr are absent from the compositions (Ref. 20). Single crystal castings are prone to all the same casting defects such as porosity and nonmetallic inclusions and control of the growth direction as the DS castings.

In addition it must be assured that the product is in fact only one crystal or at least will behave as a single crystal. The mechanism of recrystallization of superalloys is reviewed in Ref. 21. Surface recrystallization of MM 002³ is shown to be retarded by the presence of γ' (Ref. 22) or by allowing recovery at relatively high temperatures (1200 °C). It is common practice to specify that single crystal superalloys be solution heat treated immediately after casting to reduce the chance of subsequent recrystallization. In spite of such a precaution recrystallization might occur as a result of casting stresses or careless handling. An example of a recrystallized single crystal turbine blade is shown in Fig. 24 (Ref. 16). The blade shown was run in a rotating rig for about 2 hr at elevated temperature. It can be seen that a portion of the blade near the tip has separated from the airfoil. While I know of no published information on the mechanical behavior of recrystallized single crystal superalloys, it seems likely that the grain boundaries should be extremely weak. That is because

³MM is a trade name of the Martin Marietta Corporation.

single crystal alloys contain none of the elements, normally present in polycrystalline superalloys, which provide grain boundary strengthening (i.e. B, Zr).

The stress rupture life of single crystal alloys which are directionally coarsened (rafted) perpendicular to the applied stress axis are thought to be improved (Ref. 23). It has been reported that such coarsening can occur during gas turbine engine operation (Ref. 24). A more detailed examination of some of the blades from that study has been performed at the Lewis Research Center.⁴ It found that normal rafting perpendicular to the centrifugal stress axis had occurred over much of the blade. However it was further noted that some regions near the surface of the blade had rafted parallel to the blade's centrifugal stress axis, Fig. 25. The cause for this behavior is currently unknown as are the implications relative to the mechanical durability of the blade. It remains to be determined if this interesting metallographic observation is truly a defect.

CONCLUDING REMARKS

The development of the microstructures of current performance superalloys has been reviewed with emphasis on some of the microstructural defects which might be found in those alloys. It was intended to show that as superalloy chemistries and processing became more sophisticated, the nature of the defects also became more sophisticated. As the industry developed improved alloys and processes, new defects were also developed, or defects previously identified in one segment of the industry was found anew in another segment. As the superalloy industry continues its growth in technology it must remain vigilant to the prospects of also developing new defects.

⁴Private communication Mr. D.R. Hull.

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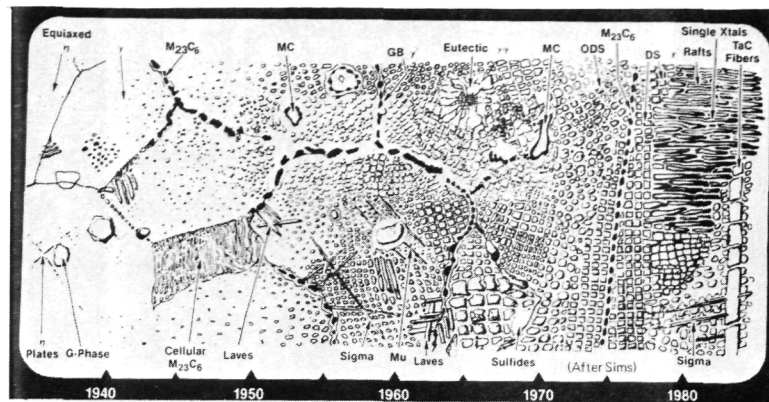


Figure 1. - The evolution of nickel-base superalloy microstructures.

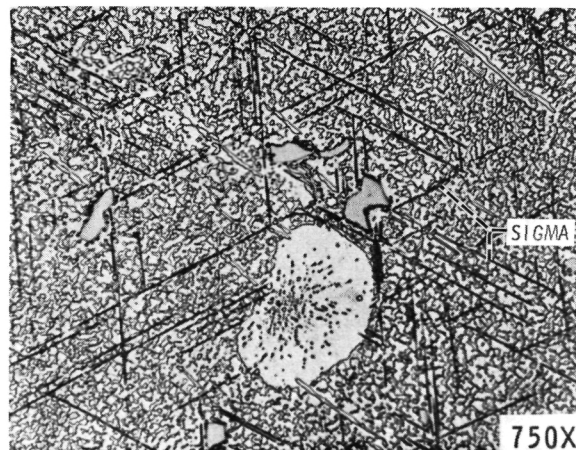


Figure 2. - Sigma phase in IN-100.

ALLOY - IN 100
EXPOSURE TEMPERATURE, 1550°F

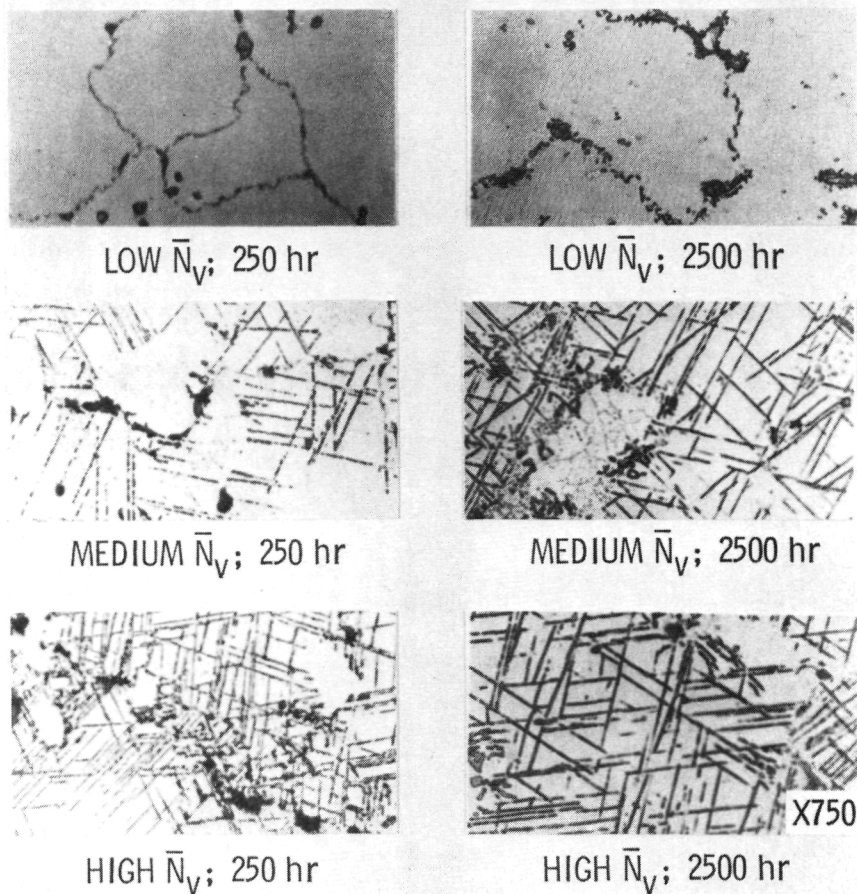


Figure 3. - Sigma amounts are a function of exposure time and composition.

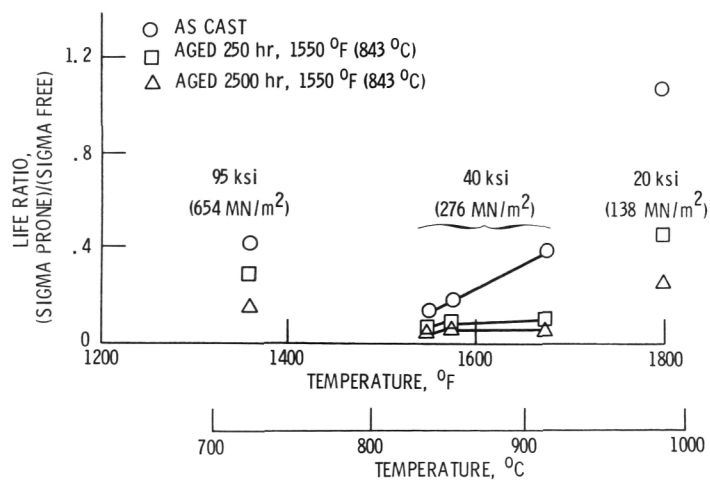


Figure 4. - Sigma precipitation can drastically reduce stress rupture life; alloy - IN 100.

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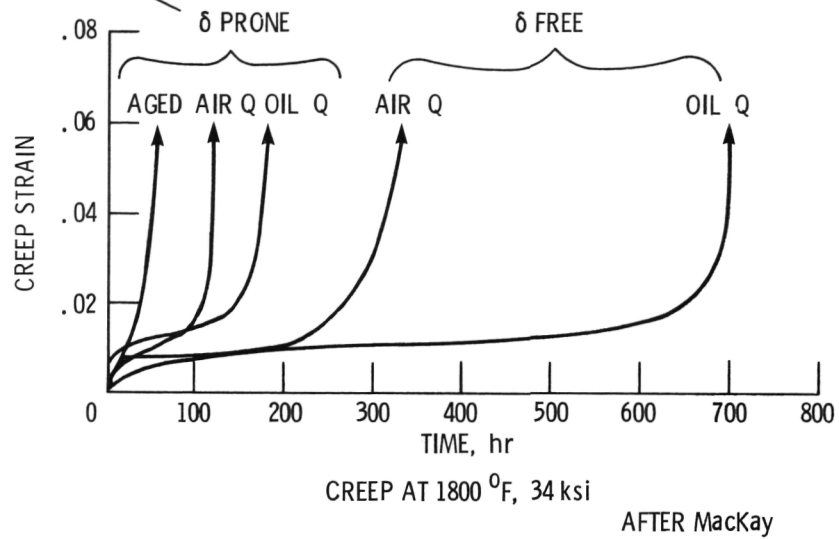
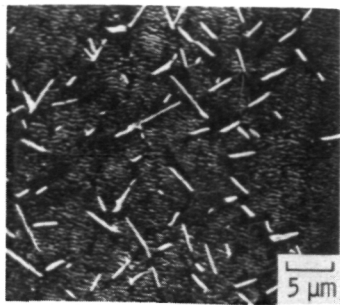


Figure 5. - Delta phase precipitation reduces creep rupture life in alloy 143 single crystals.

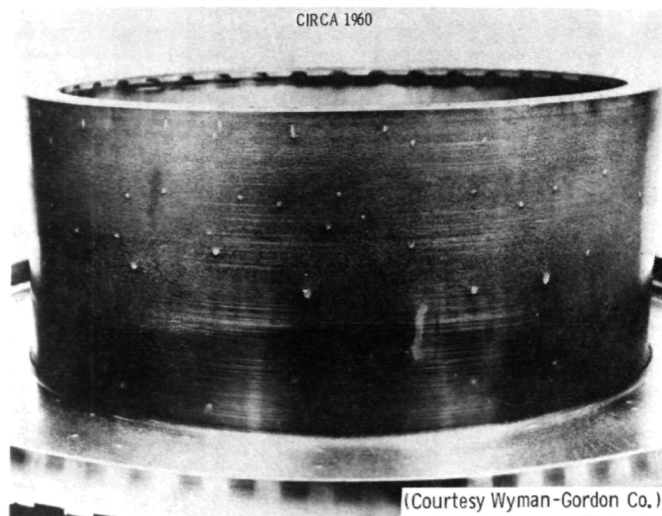


Figure 6. - Freckles in machined compressor disk.

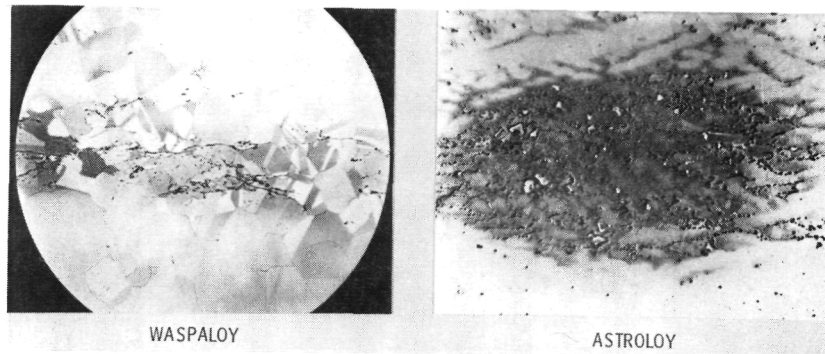


Figure 7. - Microstructures of freckles.

(Courtesy Wyman-Gordon Co.)

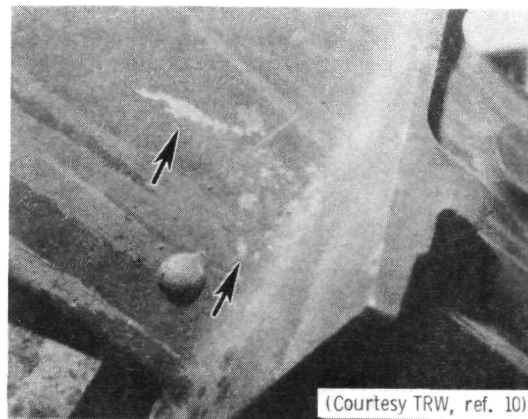


Figure 8. - Freckled DS turbine blade.

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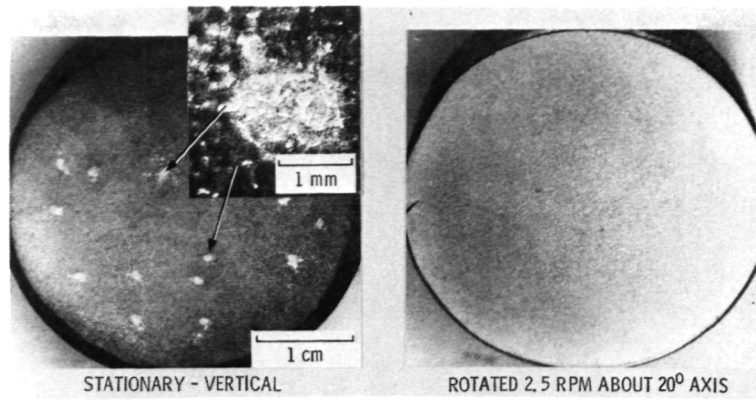


Figure 9. - Control of fluid flow can eliminate freckles in lead-tin alloy.

(Refs. 11, 12)

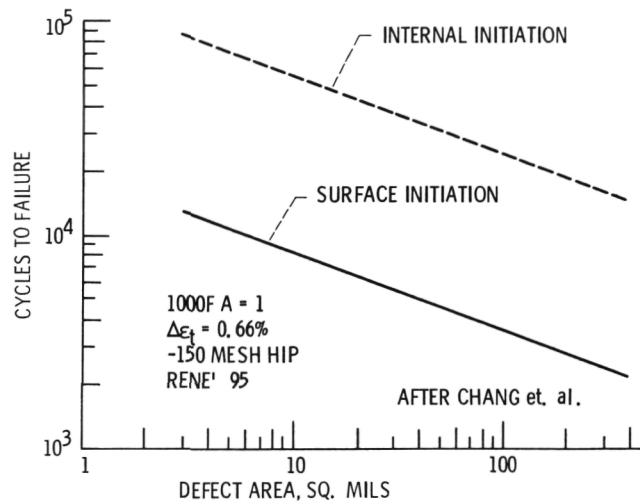


Figure 10. - Defect LCF degradation is a function of defect size and location.

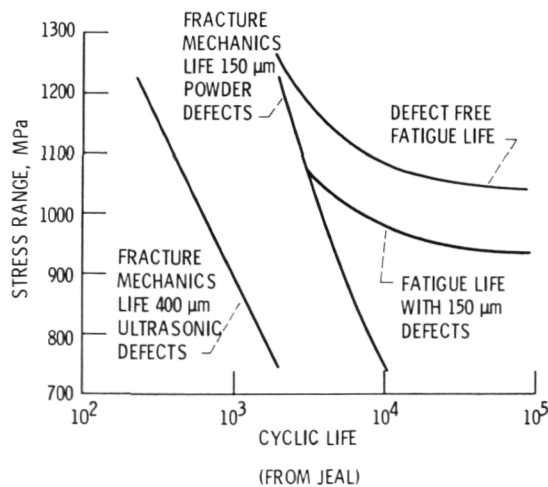


Figure 11. - PM disk design must consider defects. Cyclic fatigue behavior of P/M astroloy at 600 °C.

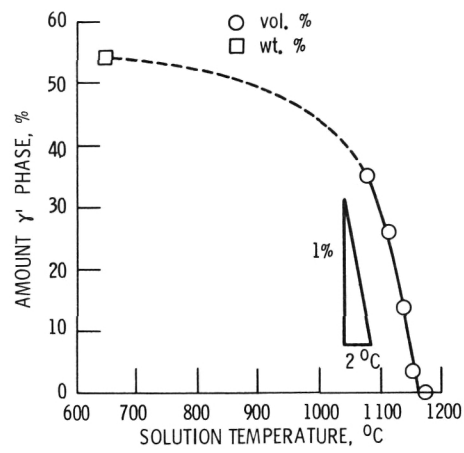


Figure 12. - The amount of undissolved γ' is very sensitive to sub-solvus temperature; René 95.

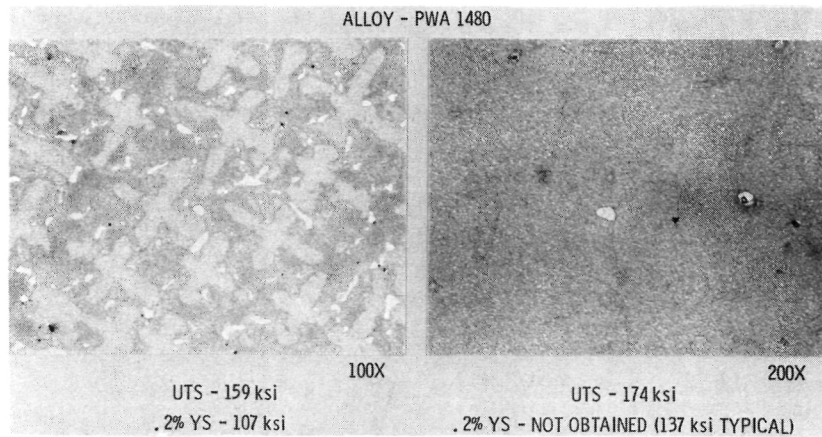


Figure 13. - Single crystal tensile strength is reduced by undissolved primary γ' .

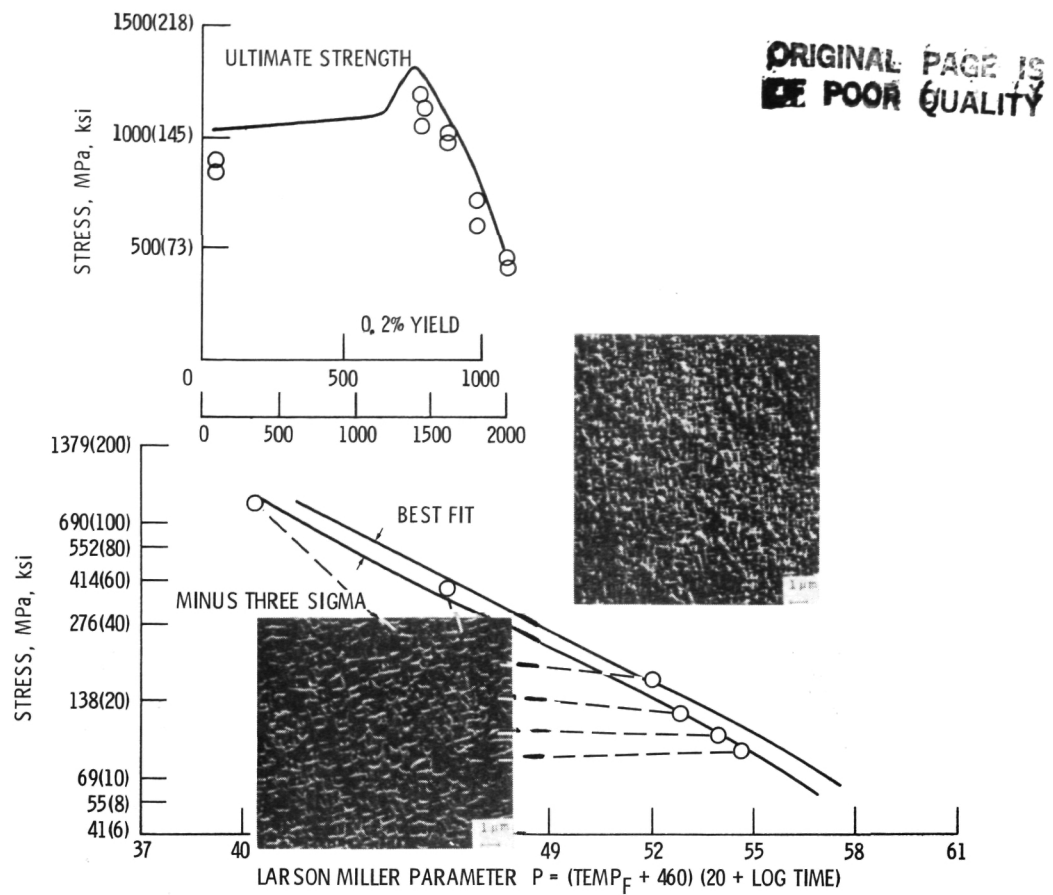


Figure 14. - Delayed quench reduces mechanical properties; alloy NASAIR 100 (ref. 16).

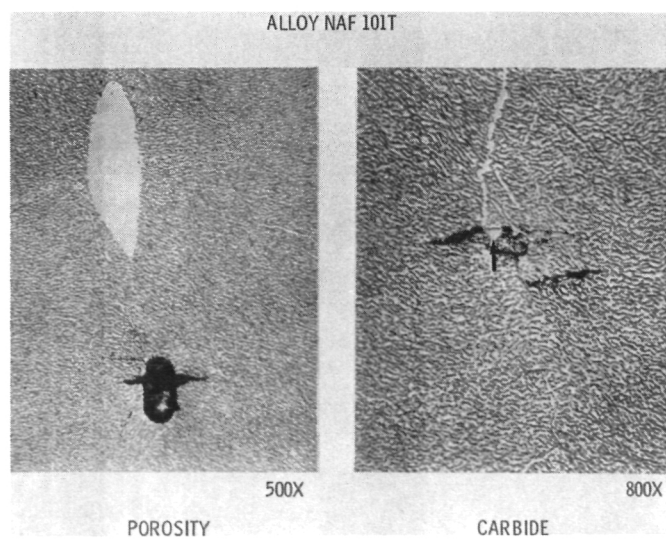


Figure 15. - Secondary stress rupture cracks in a single crystal superalloy.

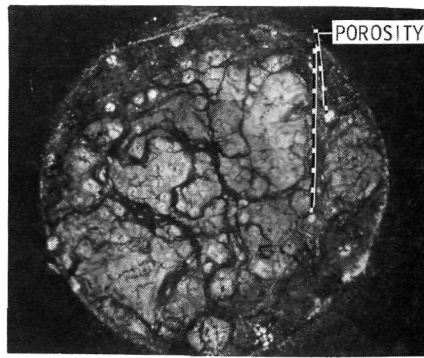


Figure 16. - Porosity in LCF fracture.

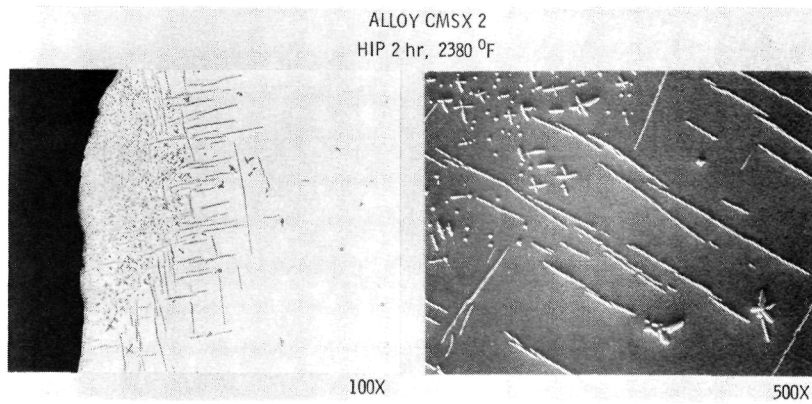


Figure 17. - Hip atmosphere can cause carburization and melting.

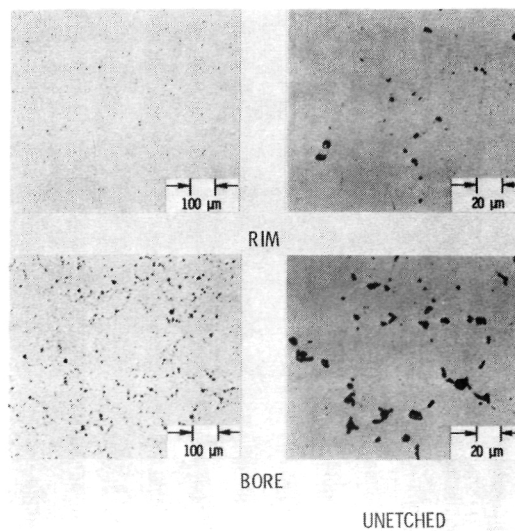


Figure 18. - Microstructure of porous LC Astroloy pressing.

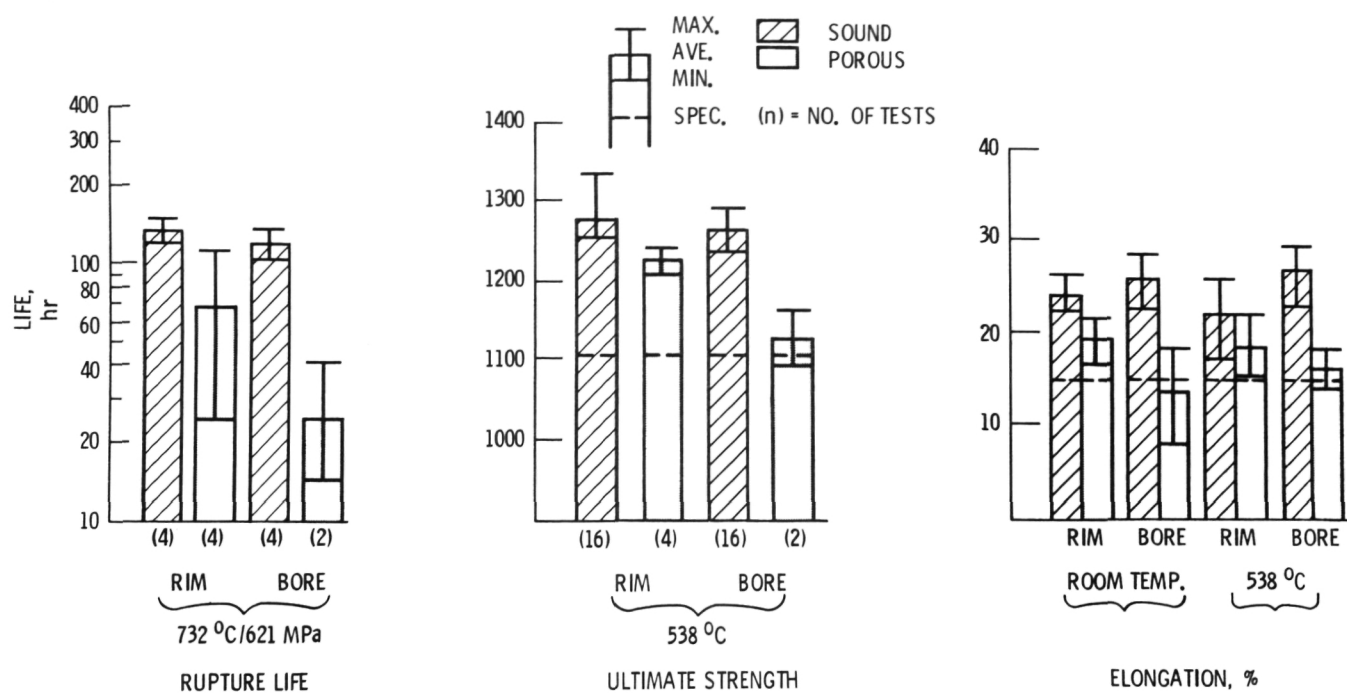


Figure 19. - Tip reduces properties of LC astroloy.

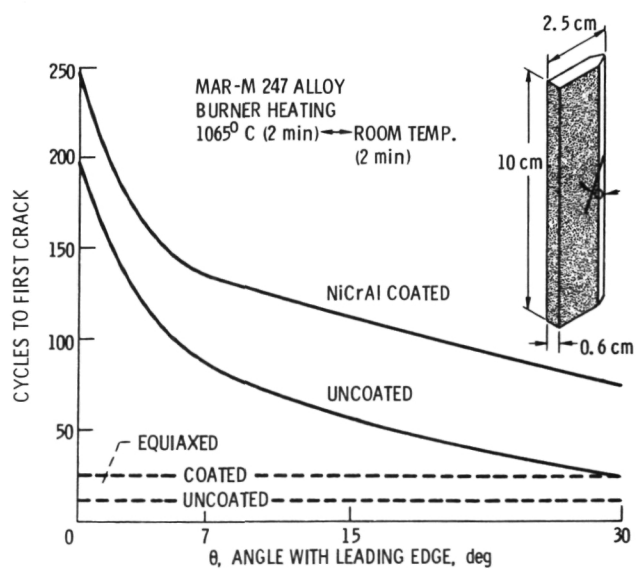


Figure 20. - Effect of DS grain orientation on thermal fatigue.

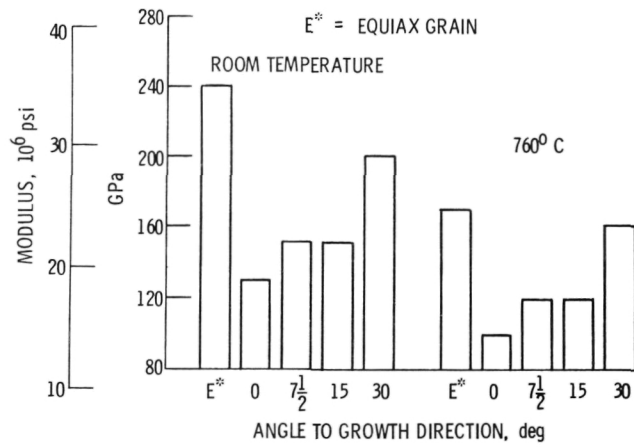


Figure 21. - Effect of DS growth direction on Young's modulus of MAR-M 247.

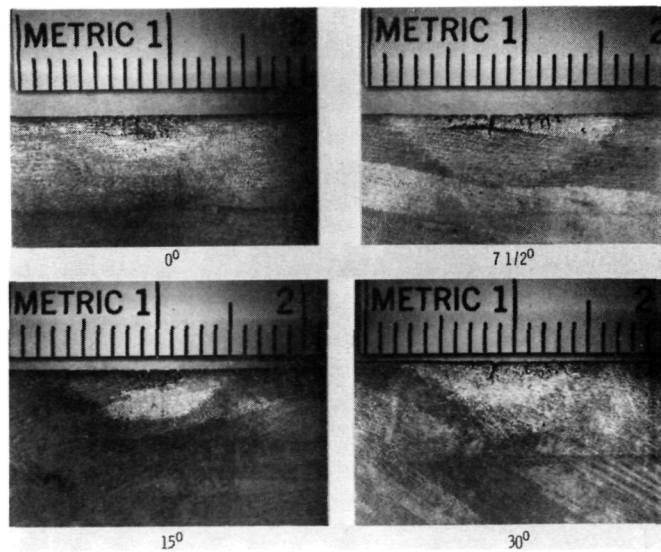


Figure 22. - Grain boundary emergent angle has little effect on thermal fatigue of DS mar M247.

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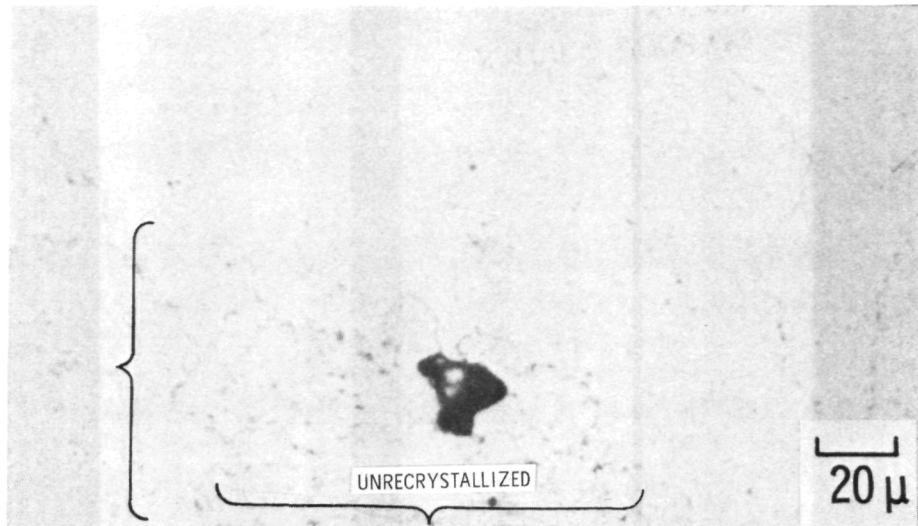


Figure 23. - Improper processing causes unrecrystallized region in MA 6000.

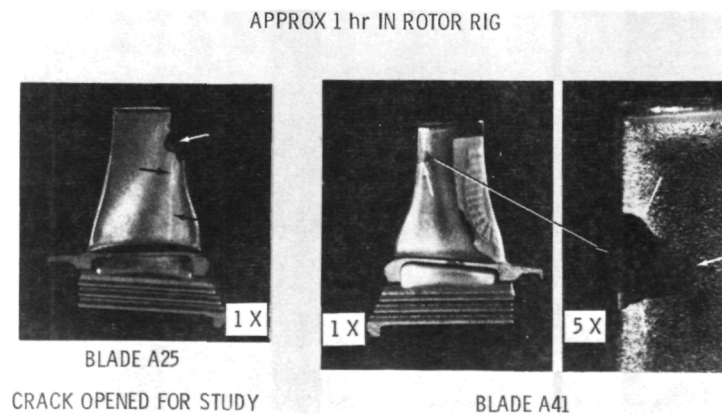


Figure 24. - Single crystal alloys will not tolerate recrystallization.

(Ref. 16)

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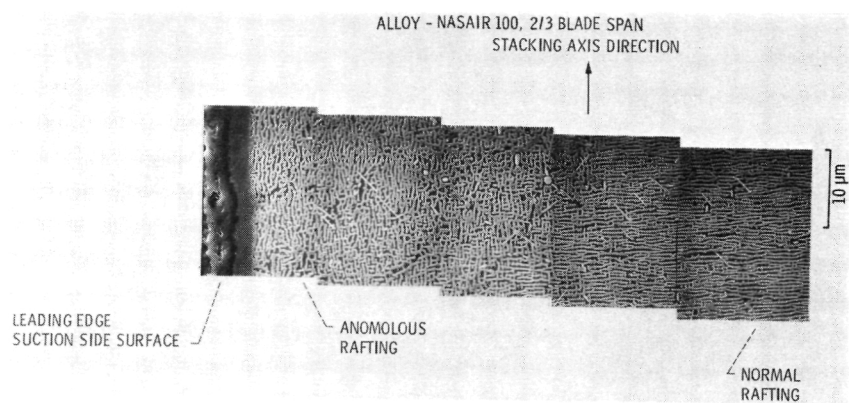


Figure 25. - γ' can raft during engine use.

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16. Abstract In the six decades since the identification of age hardenable nickel-base superalloys their compositions and microstructures have changed markedly. Current alloys are tailored for specific applications. Thus their microstructures are defined for that application. This paper will briefly review the evolution of superalloy microstructures and comment on the appearance and implications of microstructural defects in high performance superalloys. It is seen that new alloys and processes have generated new types of defects. Thus as the industry continues to develop new alloys and processes it must remain vigilant toward the identification and control of new types of defects.					
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